(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
5 July 2001 (05.07.2001)

PCT

(10) International Publication Number
WO 01/48058 A1

(51) International Patent Classification[7]: C08J 3/12, C11D 11/02, 3/37

(21) International Application Number: PCT/US99/30626

(22) International Filing Date:
22 December 1999 (22.12.1999)

(25) Filing Language: English

(26) Publication Language: English

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(81) Designated States *(national)*: AE, AL, AM, AT, AT (utility model), AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, CZ (utility model), DE, DE (utility model), DK, DK (utility model), DM, EE, EE (utility model), ES, FI, FI (utility model), GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SK (utility model), SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Designated States *(regional)*: ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:

— *With international search report.*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

WO 01/48058 A1

(54) Title: A PROCESS FOR DRYING POLYMERS

(57) Abstract: A process for drying polymers including the steps of (a) and (b). Step (a) involves making a slurry by mixing from about 0.5 % to about 20 % by weight of a surfactant; from about 30 % to about 65 % by weight of an inorganic electrolyte; and from about 30 % to about 60 % by weight of a polymer. The polymer is a copolymer of maleic acid and acrylic acid, a salt of a copolymer of maleic acid and acrylic acid and mixtures of these polymers. The formula of the polymer is H-[CH(COOM)-CH_2-]$_x$-[-CH(-COOM)-CH(-COOM)-]$_y$-H. M is a counterion and the molecular weight of the polymer is from about 5000 to about 15000. The molar ratio of x to y is from 3:7 to about 7:3. The moisture content of the slurry is from about 30 % to about 70 % by weight. Step (b) involves spray-drying the slurry to form particles. The moisture content of the particles obtained by step (b) is less than 15 % by weight of the particles.

A PROCESS FOR DRYING POLYMERS

FIELD OF THE INVENTION

The present invention relates to a process for drying polymers. Particularly, the present invention relates to a process for drying polymers for detergent compositions.

BACKGROUND OF THE INVENTION

Homopolymers and copolymers containing acrylic acid and/or maleic acid have excellent properties as ingredients for detergent compositions. For example, these polymers haves soil dispersion properties and calcium or magnesium hardness sequestration properties. Thus, the polymer has been widely used as a thickening agent, a flocculating agent, a dehydrating agent, a soil conditioning agent and the like.

These polymers are usually provided in a liquid form. However, it is usually required to remove the moisture from the polymers before they can be incorporated in dry laundry products such as granules. The conventional way to incorporate polymers into dry laundry products is as follows: the liquid polymer is mixed with other detergent ingredients such as surfactants and builders to make a detergent slurry; and then the slurry is dried by a spray drying process. This approach requires substantial drying capacity that negatively impacts the detergent manufacturing economics. But dried polymers can be added to

detergents sfter the spray drying process, which helps reduce the drying load and simplifies the detergent making process. Thus, there is a continuing need for processes for drying polymers.

Processes for drying polymers using line bed dryers are known. However, the particles which are obtained by these dryers are not economically efficient because of the slow drying rate and the equipment requires a substantial amount of space. Further, if the polymers are dried alone the particles obtained are hygroscopic which pick up moisture easily. Then the particles become too sticky to handle in future manufacturing steps.

Thus, there is a continuing need for polymer drying processes that do not use line bed dryers.

SUMMARY OF THE INVENTION

The present invention relates to a process for drying polymers. The process comprising the steps: (a) making a slurry; (b) spray-drying the slurry to form particles. In step (a), the slurry is made by mixing from about 0.5% to about 20% by weight of a surfactant, from about 30% to about 65% by weight of an inorganic electrolyte; and from about 30% to about 60% by weight of a polymer. The polymer is selected from the group consisting of a copolymer of maleic acid and acrylic acid, a salt of a copolymer of maleic acid and acrylic acid and mixtures thereof. The formula of the polymer is as follows:

H-[CH(COOM)-CH_2-]$_x$-[-CH(-COOM)-CH(-COOM)-]$_y$-H,

wherein M is a sodium or potassium, the molecular weight of the polymer is from about 5000 to 15000, and the molar ratio of x to y is from 3:7 to 7:3. In step (a), the moisture content of the slurry is from about 30% to about 70% by weight. After step (b), the moisture content of the particles is less than 15% by weight of the particles.

The processes of the present invention require a spray-drying step. Spray-drying process can provide many benefits including: the ability to control particle size; economical drying; small scale equipment; more efficient drying; rounder particles.

DETAILED DESCRIPTION OF THE INVENTION

While the specification concludes with claims particularly pointing out and distinctly claiming the invention, it is believed the present invention will be better understood from the following description.

All percentages are by weight of total composition unless specifically stated otherwise.

All ratios are weight ratios unless specifically stated otherwise.

Definitions

As used herein, "comprising" means that other steps and other ingredients which do not affect the end of result can be added. This term encompasses the terms "consisting of" and "consisting essentially of".

All cited references are incorporated herein by reference in their entireties. Citation of any reference is not an admission regarding any determination as to its availability as prior art to the claimed invention.

The process of the present invention

(a) making a slurry

The first step of the processes of the present invention is to make a slurry. The slurry is made by mixing a surfactant, an inorganic electrolyte, and a polymer. Herein, "slurry" refers to a mixture of a detergent surfactant and a builder, wherein the mixture is substantially non-solid in form. The slurry can also include other ingredients such as brighteners and buffers.

(1) Surfactant

A surfactant is an essential ingredient for the step (a). The surfactant is preferably selected from the group consisting of anionic surfactants, zwitterionic surfactants, ampholytic surfactants, cationic surfactants, and mixtures thereof. More preferably, the surfactant is selected from the group consisting of anionic

surfactants and mixtures thereof. Even more preferably, the surfactant is selected from the group consisting of Linear Alkylbenzene Sulfate (LAS), Alkyl Sulfate (AS) and mixtures thereof. Most preferably, the surfactant is a mixture of LAS and AS where the ratio of LAS to AS is from about 2:1 to about 1:2. Preferably the slurry contains from about 0.5% to about 20% by weight, preferably, from about 1% to about 15% of the surfactant. The surfactant of the present invention also can be selected from description of WO 92/06154 to Cook, et al., published on April 16, 1992 which is incorporate herein by reference.

(2) Inorganic Electrolyte

An Inorganic electrolyte is an essential ingredient for step (a).

The inorganic electrolyte is preferably selected from the group consisting of sodium carbonates, potassium carbonates, sodium sulphates, potassium sulphates, sodium silicates, potassium silicates and mixtures thereof. Preferably, the inorganic electrolyte is selected from the group consisting of sodium carbonates, potassium carbonates and mixtures thereof. Preferably the slurry contains from about 30% to about 65% by weight, preferably, from about 30% to about 50% of the inorganic electrolyte.

Examples of carbonate builders are the alkaline earth and alkali metal carbonates as disclosed in German Patent Application No. 2,321,001 to Jacobsen, et al., published November 15, 1973 which is incorporate herein by reference.

(3) Polymer

A polymer is an essential ingredient for step (a). The polymer is selected from the group consisting of a copolymer of maleic acid and acrylic acid, a salt of a copolymer of maleic acid and acrylic acid and mixtures thereof. The formula of the polymer is as follows:

H-[CH(COOM)-CH_2-]$_x$-[-CH(-COOM)-CH(-COOM)-]$_y$-H,

wherein M is a counterion, preferably sodium or potassium, the molecular weight of the polymer is from about 5000 to about 15000, preferably, from about 6000 to about 12000, and the molar ratio of x to y is from about 3:7 to about 7:3, preferably, from about 1:1 to about 7:3. The slurry contains from about 30% to about 70%, preferably, from about 40% to about 60%, by weight of the polymer. The polymer of the present invention also can be selected from descriptions of WO 9533815, Murata, et al., published December 14, 1995, WO 91/08281 to Foster and Vandepas, published June 13, 1991 and PCT 90/01815 to Bieringer and Steinhardt, published February 22, 1990 which are incorporate herein by reference.

(4) Moisture

The slurry in step (a) should contain a liquid. The liquid, or moisture may come from the surfactants and the polymers described above because they are usually provided as a liquid or a paste. The moisture content of the slurry is from about 30% to about 70% by weight.

(b) spray-drying

A second step of the process of present invention is to spray-dry the slurry produced in step (a). Step (b) preferably includes dispersing the slurry, which is preferably aqueous, under pressurized nuzzles or disc atomizer in spray drying tower through which hot gases are counter- or co-currently blowing. This step can be carried out in conventional spray drying equipment such as a conventional tower as well as other spray drying apparatus. Conventional spray drying equipment may include, for example, a spray-dryer which is provided by Ohkawara. Co., Ltd. (Japan) or Niro Co., Ltd. (Denmark). For drying detergent

granules conventional spray drying equipment usually has a height of about 0.5m to about 30m and an inside temperature of from about 200°C to about 500°C.

Examples of the spray-drying process of the present invention are described in U. S. Patent 5149455, Jacobs et al, issued September 22, 1992, and U. S. Patent 5565442, Del Greco et al, issued October 15, 1996 which are incorporate herein by reference.

Optional steps

The spray-dried granules, can be used in detergent compositions or they can be further processed by a compacting machine. Spray-dried granules from a tower can also can be densified further by loading a liquid such as water or a nonionic surfactant into the pores of the granules or subjecting them to one or more high speed mixer/densifiers. A suitable high speed mixer/densifier for this process is a device marketed under the tradename "Lödige CB 30" or "Lödige CB 30 Recycler" which comprises a static cylindrical mixing drum having a central rotating shaft with mixing/cutting blades mounted thereon. In use, the ingredients for the composition are introduced into the drum and the shaft/blade assembly is rotated at speeds in the range of 100-2500 rpm to provide thorough mixing/densification. See U.S. Patent 5,149,455, Jacobs et al, issued September 22, 1992, and U.S. Patent 5,565,422, Del Greco et al, issued October 15, 1996. Other such apparatus includes the devices marketed under the tradename "Shugi Granulator" and under the tradename "Drais K-TTP 80".

Another process step which can be used to further densify spray-dried granules involves treating the spray-dried granules in a moderate speed mixer/densifier. Equipment such as that marketed under the tradename "Lödige KM" (Series 300 or 600) or "Lödige Ploughshare" mixer/densifiers are suitable for this process step. Such equipment is typically operated at 40-160 rpm. The residence time of the detergent ingredients in the moderate speed mixer/densifier is from about 0.1 to 12 minutes conveniently measured by dividing the steady state mixer/densifier weight by the throughput (*e.g.*, Kg/hr). Other useful equipment includes the device which is available under the tradename "Drais K-T

160." This process step which employs a moderate speed mixer/densifier (*e.g.*, Lödige KM) can be used by itself or sequentially with the aforementioned high speed mixer/densifier (*e.g.*, Lödige CB) to achieve the desired density. Other types of granules manufacturing apparatus useful herein include the apparatus disclosed in U.S. Patent 2,306,898, G. L. Heller, issued December 29, 1942.

While it may be more suitable to use the high speed mixer/densifier followed by the low speed mixer/densifier, the reverse sequential mixer/densifier configuration also can be used. One or a combination of various parameters including residence times in the mixer/densifiers, operating temperatures of the equipment, temperature and/or composition of the granules, the use of adjunct ingredients such as liquid binders and flow aids, can be used to optimize densification of the spray-dried granules in the process of the invention. By way of example, see the processes in U.S. Patent 5,133,924, Appel et al, issued July 28, 1992; U.S. Patent 4,637,891, Delwel et al, issued January 20, 1987; U.S. Patent 4,726,908, Kruse et al, issued February 23, 1988; and, U.S. Patent 5,160,657, Bortolotti et al, issued November 3, 1992.

Furthermore, detergent compositions according to the invention can be produced by blending conventional or densified spray-dried detergent granules with detergent agglomerates in various proportions (*e.g.*, a 60:40 weight ratio of granules to agglomerates) produced by one or a combination of the processes discussed herein. See U.S. Patent 5,569,645, Dinniwell et al, issued October 29, 1996. Additional adjunct ingredients such as enzymes, perfumes, brighteners and the like can be sprayed or admixed with the agglomerates, granules or mixtures thereof produced by the processes discussed herein.

EXAMPLES

The following examples further describe and demonstrate embodiments within the scope of the present invention. The examples are given solely for the purpose of illustration and are not to be construed as limitations of the present invention, as may variations thereof are possible without departing from the spirit and scope of the present invention. Where applicable, ingredients are identified by chemical name, or otherwise defined below.

LAS	Sodium linear alkyl benzene sulphonate
AS	Alkyl Sulphate
MA	Maleic acid
AA	Acrylic acid
Polymers	A copolymer of maleic acid and acrylic acid

Example A-E

These formulations are examples of particles containing detergent surfactants, inorganic electrolytes, polymers and moistures.

	A	B	C	D	E
LAS	5	-	5	6	3
AS		9		6	2
Sodium carbonate	45	45		30	
Sodium Sulfate		-	45		
Potassium carbonate					45
Polymer (MA:AA)	45	45	45	55	45
Miscellaneous (moisture, unreacted)	5	1	5	3	5
Total	100	100	100	100	100

The above described composition are suitably made as follows:

1. A slurry comprising the surfactant, the inorganic electrolyte and polymer is prepared and mixed in a crutcher at 50-80°C.
2. The slurry is dried by a conventional spray dryer (Niro) with an inlet air temperature of between 220-350°C, and outlet temperature of about from 80-120°C. The slurry is atomized by pressure nozzles, disc atomizer or other types of spray dryers.
3. The dried base powder exits the spray drying tower at temperature of 70-100°C and can be cooled down via an airlift to 20-40°C.
4. The dried base powder has a density of about 100g/L to 500g/L

The base powder prepared by this process had good physical properties, good flowability, good stability (does cake/lump easily), and can be directly used as a raw material for any downstream detergent process with no/minimal modification.

The embodiments disclosed and represented by the previous examples have many advantages. For example, the particle prepared by the present invention have improved physical properties such as flowability and stability.

It is understood that the foregoing detailed description of the examples and embodiments of the present invention are given merely by way of illustration, and that numerous modifications and variations may become apparent to those skilled in the art without departing from the spirit and scope of the invention; and such apparent modifications and variations are to be included in the scope of the appended claims.

WHAT IS CLAIMED IS:

1. A process for drying polymers comprising the steps of:

(a) making a slurry by mixing

(i) from about 0.5% to about 20% by weight of a surfactant

(ii) from about 30% to about 65% by weight of an inorganic electrolyte; and

(iii) from about 30% to about 60% by weight of a polymer,

wherein the polymer is selected from the group consisting of a copolymer of maleic acid and acrylic acid, a salt of a copolymer of maleic acid and acrylic acid and mixtures thereof, and wherein the formula of the polymer is

H-[CH(COOM)-CH_2-]$_x$-[-CH(-COOM)-CH(-COOM)-]$_y$-H,

wherein M is a counterion, the molecular weight of the polymer is from about 5000 to about 15000, and the molar ratio of x to y is from 3:7 to about 7:3 and wherein the moisture content of the slurry is from about 30% to about 70% by weight ; and

(b) spray-drying the slurry to form particles;

wherein the moisture content of the particles obtained by step (b) is less than

15% by weight of the particles.

2. A process according to Claim 1, wherein the surfactant is selected from the group consisting of anionic surfactants, zwitterionic surfactants, ampholytic surfactants, cationic surfactants, and mixtures thereof.

3. A process according to Claim 1, wherein the surfactant is an anionic surfactant selected from the group consisting of linear alkylbenzene sulfonate, alkyl sulfate and mixtures thereof.

4. The process according to Claim 1, wherein the molecular weight of the polymer is from about 6000 to 12000 and the molar ratio of x to y is from 1:1 to 7:3.

5. The process according to Claim 1, further comprising a step of compacting the particles produced in step (b).

6. The process according to Claim 1, wherein step (b) is conducted at a temperature of from about 220 °C to about 350°C.

7. The process according to step (b) in Claim 1, wherein the moisture content of the particles produced in step (b) is from about 1% to about 15%.

8. The particles made according to the process of any of claims 1 to 6.

9. The particle according to Claim 8, wherein the particles have a particle size distribution of from about 50 micrometers to about 500 micrometers.

10. A detergent composition comprising the particle according to Claim 8.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 99/30626

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C08J3/12 C11D11/02 C11D3/37

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C08J C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, PAJ, EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4 900 466 A (ATKINSON COLIN ET AL) 13 February 1990 (1990-02-13) claims 1,2 column 5, line 20 - line 68	1
A	EP 0 240 356 A (UNILEVER PLC ;UNILEVER NV (NL)) 7 October 1987 (1987-10-07) claims 1,4-6 example 4; table page 3, line 45 - line 46 page 4, line 52 - line 56	1
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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

* Special categories of cited documents :

A document defining the general state of the art which is not considered to be of particular relevance

E earlier document but published on or after the international filing date

L document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

O document referring to an oral disclosure, use, exhibition or other means

P document published prior to the international filing date but later than the priority date claimed

T later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

X document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

Y document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

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Date of the actual completion of the international search	Date of mailing of the international search report
6 September 2000	15/09/2000

Name and mailing address of the ISA	Authorized officer
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Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 99/30625

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 453 215 A (KARPUSIEWICZ WILLIAM M ET AL) 26 September 1995 (1995-09-26) claim 1	1
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INTERNATIONAL SEARCH REPORT

Information on patent family members

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